Designation: ~~D5582 – 14~~ D5582 – 22

Standard Test Method for Determining Formaldehyde Levels from Wood Products Using a Desiccator[1]

This standard is issued under the fixed designation D5582; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a small scale procedure for measuring formaldehyde emission potential from wood products under defined test conditions. The formaldehyde level is determined by collecting air-borne formaldehyde in a small distilled water reservoir within a closed desiccator. The quantity of formaldehyde is determined by a modification of the National Institute for Occupational Safety and Health (NIOSH) 3500 chromotropic acid test procedure. Other analytical procedures may be used to determine formaldehyde emission potential provided that such methods give similar results to the chromotropic acid procedure. However, the test results and test report must be properly qualified and the analytical procedure employed must be noted. Procedures based on acetylacetone and pararosaniline have been found to give similar results to chromotropic acid in other test methods used in determining formaldehyde emission potential from wood products (see Test Method E1333).

1.2 Wood products typically evaluated by this test method are made with urea-formaldehyde adhesives and include ~~particleboard, hardwood,~~particle-board, hardwood plywood, and medium-density fiberboard. This test method is used for product quality control and is a small bench test method that correlates with the large-scale acceptance test for determining formaldehyde levels from wood products, Test Method E1333. Alternative conditioning intervals may give better correlation, such as seven day conditioning that parallels Test Method E1333. The general desiccator testing procedure may be modified for different conditioning times to accommodate its use in manufacturing quality control. However, the test results must be properly qualified and the conditioning time employed must be noted.

NOTE 1—If modifications are made to the conditioning period for quality control purposes, it is important that the modification is consistently applied. Otherwise, the results may not be comparable.

1.3 The values stated in SI units are to be regarded as ~~the~~ standard. The values given in parentheses ~~are for information only.~~after SI units are provided for information only and are not considered standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*For specific hazard statements, see Section 6 and 8.2.5.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

[1] This test method is under the jurisdiction of ASTM Committee D07 on Wood and is the direct responsibility of Subcommittee D07.03 on Panel Products.

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

E77 Test Method for Inspection and Verification of Thermometers

E337 Test Method for Measuring Humidity with a Psychrometer (the Measurement of Wet- and Dry-Bulb Temperatures)

E1333 Test Method for Determining Formaldehyde Concentrations in Air and Emission Rates from Wood Products Using a Large Chamber

2.2 *HUD Document:*

24 CFR 3280, Manufactured Home Construction and Safety Standards, Federal Register, Vol 49, No. 155[3]

2.3 *NIOSH Document:*

Formaldehyde Method 3500, U.S. Department of Health, and Human Services[3]

2.4 *Other Documents:*

Minnesota Statutes Section 144.495, 325F.18, and 325F.181, Formaldehyde Gases in Building Materials[4]

California Air Resources Board (CARB), California Code of Regulations Sections 93120-93120.12, Title 17 Airborne Toxic Control Measure to Reduce Formaldehyde Emissions from Composite Wood Products[5]

EPA TSCA Title VI 40 CFR Section 770, Formaldehyde Standards for Composite Wood Products[6]

3. Significance and Use

3.1 Limitations have been established on formaldehyde emission levels for wood panel building products made with urea-formaldehyde adhesives and permanently installed in homes or used as components in kitchen cabinets and for similar industrial products. This test method is used in conjunction with the test method referenced by HUD Rules and Regulations 24 CFR 3280 for manufactured housing, California Air Resources Board (CARB) regulation 93120, EPA TSCA Title VI 40 CFR Section 770, and by Minnesota Statutes Section 144.495 for housing units and building materials. This test method provides a means of testing small-size samples to determine formaldehyde emission potential.

3.2 This test method incorporates a desiccator, with the desiccant removed, having a ~~250-mm (10-in.)~~ 250 mm (10 in.) inside diameter and a volume of approximately 10.5 L (641 in.^{3}) with the desiccator lid in place. Conditions controlled in the procedure are as follows:

3.2.1 Conditioning of panel products prior to testing,

3.2.2 Specified number, size, and edge sealing of wood specimens to be placed in the desiccator,

3.2.3 Test desiccator temperature, and

3.2.4 Samples from the ~~25-mL~~ 25 mL distilled water collection medium in the petri dish bottom are analyzed for formaldehyde at the end of a 2-h period in the closed desiccator.

3.3 This test method employs a single set of environmental conditions to assess formaldehyde emission potential from certain wood products. When the relationship between desiccator test values and large-chamber test values are to be determined, the values for the specific wood panel product type shall be plotted. This test method does allow a comparison of formaldehyde levels from different products for the same use.

~~NOTE 2—Care must be exercised in the extension of the results to actual formaldehyde emission from products under actual use conditions.~~

3.3.1 Care must be exercised in the extension of the results to actual formaldehyde emission from products under actual use conditions.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Standardization Documents Order Desk, DODSSP, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5098, http://www.dodssp.daps.mil.

[4] Available from Print Communications, Dept. of Administration, 117 University Ave., St. Paul, MN 55155.

[5] Available from California EPA website: http://www.arb.ca.gov/toxics/compwood/compwood.htm.

[6] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

4. Interferences

4.1 The NIOSH 3500 analytical method lists phenols as a negative interference when present at an 8:1 excess over formaldehyde. Modifications in the analytical procedure shall be made when this test method is used to accurately determine the formaldehyde emission potential from wood products made with phenol-formaldehyde adhesive systems.[7, 8]

5. Apparatus

5.1 *Desiccator*—The interior volume of the desiccator shall be 10.5 L (641 in.^{3}). Any desiccant shall have been removed, the interior of the desiccator thoroughly cleaned, and the porcelain desiccator plate replaced in the desiccator. The bearing areas of the desiccator and desiccator lid shall be greased so that the container will be air tight during the duration of the 2-h test.

5.2 *Petri Dish and Beaker*—A clean ~~400-mL~~ 400 mL beaker to be inverted as a reservoir support and the bottom of a 100 mm by ~~20-mm~~ 20 mm petri as a distilled water reservoir dish shall be available for each desiccator test.

5.3 *Test Room or Area*—A room or test area capable of being maintained at ~~24~~24 °C ± ~~1°C (75~~1 °C (75 °F ± ~~2°F)~~2 °F) shall be available for conducting desiccator tests.

NOTE 2—If liquid-in-glass thermometers are used for determining or checking the temperature of the test area, see Test Method E77.

5.4 Examples of acceptable reagents, materials, and equipment are provided in Appendix X1.

6. Hazards

6.1 *Chromotropic Acid Reagent Treatment (see 8.2.4 and A3.5)*—During this hazardous operation, the operator shall wear rubber gloves, apron, and a full face mask or be protected from splashing by a transparent shield such as a hood window. The solution becomes extremely hot during the addition of sulfuric acid. Add slowly to avoid loss of sample due to splattering.

6.2 *Cleaning Chemicals for Glassware*—Appropriate precautions shall be taken if cleaning chemicals are considered to be hazardous.

7. Test Specimens

7.1 Use eight 70 mm ± 2 mm by 127 mm ± ~~2-mm~~ 2 mm (2¾ in. ± 0.08 in. by 5 in. ± 0.08 in.) by panel thickness specimens for each desiccator test. Cut specimens from the sample panel or panel segment to obtain adequate representation of areas within the panel or panel segment. The fresh cut edges and ends of each specimen shall be at least 25 mm (1 in.) from the edges and ends of the sample panel or panel segment. When a product has significantly different emission characteristics for each surface and has only one surface exposed to the building space, also use sixteen 70 mm ± 2 mm by 127 mm ± 2 mm (2¾ in. ± 0.08 in. by 5 in. ± 0.08 in.) test pieces to prepare eight double-piece back-to-back specimens.

7.2 *Specimen Edge Sealing*—Remove sawdust and loose splinters from each test specimen. Coat the edges and ends of each single or double-piece specimen by immersion in melted paraffin wax. Apply at least two coats. The wax shall cover no more than 5 mm (3⁄16 in.) of either face around the coated perimeter.

7.3 *Specimen Conditioning*—Then condition the specimens on edge, spaced apart, so air can freely circulate across all surfaces for seven days ±4 h at ~~24~~24 °C ± ~~1°C (75~~1 °C (75 °F ± ~~2°F)~~2 °F) and 50 ± 10 % relative humidity. The formaldehyde concentration in the air within 30 cm (12 in.) of where the specimens are conditioned shall be not more than 0.04 ppm during the conditioning period.

NOTE 3—Conditioning time less than seven days and specimens with edges and ends not coated with paraffin wax may be used for quality control or informational testing; however these and other test method modifications shall be clearly indicated in the test report. Modifications to conditioning time or edge treatment, or both, will affect the test results; therefore, correlation to other test methods may need to be re-established.

[7] Hakes, D., Johnson, G., and Marhevka, J., *Procedure for Elimination of Phenol Interference in the Chromotropic Acid Method for Formaldehyde*, American Industrial Hygiene Association, April 1984.

[8] *Technical Bulletin No. 415*, National Council of the Paper Industry for Air and Stream Improvement Inc. (NCASI), 1983.

NOTE 4— If liquid-in-glass thermometers or psychrometers, or both, are used for determining or checking the temperature or the relative humidity, or both, of the conditioning area, see Test Methods E77 and E337.

8. Procedure

NOTE 5—A list of test apparatus and chemical reagents are provided in Appendix X1.

8.1 *Test Procedure for Materials:*

8.1.1 Conduct tests in a room maintained at ~~24~~24 °C ± ~~0.6°C (75~~1 °C (75 °F ± ~~1°F).~~2 °F). Equilibrate the desiccator, petri dish bottom, and distilled water to room conditions.

8.1.2 Before each test, wipe the desiccator with a clean cloth or paper towel moistened with distilled water, and then dry with a clean dry cloth or paper towel.

~~NOTE 7—Formaldehyde can be used as a constituent of wet-strength resins for paper and of permanent-press resins for fabrics. The type of cloth or paper towel selected for cleaning must be formaldehyde-free.~~

8.1.2.1 Formaldehyde can be used as a constituent of wet-strength resins for paper and of permanent-press resins for fabrics. The type of cloth or paper towel selected for cleaning must be formaldehyde-free.

8.1.3 Apply a light coating of vacuum grease to the desiccator lid and desiccator. Avoid excessive use of vacuum grease.

8.1.4 Arrange specimens as prepared in 7.1 and 7.2 and condition as in 7.3 on top of the porcelain desiccator plate around an inverted ~~400-mL~~ 400 mL beaker as a 100 mm ± ~~7-mm~~ 7 mm (4 in. ± ~~¼-in.)~~ in.) high support inside the desiccator for the petri dish bottom distilled water reservoir. Specimens should be arranged so that air has access to all surfaces and edges. To obtain an empty desiccator reading, test one desiccator without any test specimens. An empty desiccator reading greater than 0.05 µg/mL indicates that the test system has been contaminated and the test results shall be voided for all related samples in the test process.

8.1.5 Pipet 25 mL of distilled water into the bottom portion of petri dish.

8.1.6 Carefully lower the petri dish bottom containing distilled water into the desiccator until it rests upon the inverted ~~400-mL~~ 400 mL beaker.

8.1.7 Slide the desiccator lid into place making sure a good seal is obtained.

8.1.8 Observe and record the time.

8.1.9 ~~Maintain~~Record the desiccator test room ~~at 24 ± 0.1°C (75 ± 2°F). Record the~~ temperature at 30-min intervals. Alternatively, use a continuous temperature recorder. Report any temperature range deviations.

8.1.10 After 120 ± 1 min, remove the desiccator lid and carefully remove the petri dish. Proceed immediately to 8.2.1. When running multiple desiccator tests, initiate 8.2.1 within 10 min, otherwise cover the petri dish or dishes with parafilm while awaiting analysis.

8.2 *Analysis of Water Samples:*

8.2.1 Gently swirl the petri dish and pipet 4 mL of the solution into each of two 16 mm by ~~150-mm~~ 150 mm screw cap test tubes for duplicate analysis. Label to avoid subsequent error. Alternatively, use three tubes for triplicate analysis.

8.2.2 Pipet 4 mL of distilled water into a 16 mm by ~~150-mm~~ 150 mm screw capped test tube to act as a "blank."

8.2.3 Add 0.1 mL of 1 % chromotropic acid reagent to each test tube and shake to mix.

8.2.4 Slowly and carefully pipet 6.0 mL concentrated sulfuric acid into each test tube (~~**Precaution**~~**Warning**—See 6.1.) and allow to flow down the side of test tube. Allow the volumetric pipet to drain. **Do not blow out**. Before placing caps on test tubes, check the condition of the polytetrafluoroethylene (PTFE) cap liners to make sure they are clean and not deteriorated.

8.2.5 Ensure adequate mixing by use of a vibrating laboratory mixer or other means. Mixing is complete when there is no sign of stratification. If absorbance readings routinely exceed 1.0 or if spectrophotometric analysis is performed within 2 h, heat capped test tubes to 95°C or place in a boiling water bath for 15 ± 2 min to ensure that the chemical reaction is complete. After removal, allow the test tubes to cool to room temperature. Carefully vent test tubes to release pressure. (**Warning**—Avoid rapid mixing as heating and pressure will increase and potentially break the test tube.)

8.2.6 Allow the tubes to cool to room temperature. Do not accelerate the cooling. Avoid cooling tubes in direct sunlight as this may alter color chromogen development. Transfer the solution to cuvettes (if necessary). At this point, small bubbles may be rising through the solution. Do not make absorbance readings until the solution is clear.

8.3 *Absorbance Readings:*

8.3.1 Prior to performing this test method for the first time, a calibration curve shall be developed. See Annex A3.

8.3.2 Standardize the spectrophotometer using distilled water at 580 nm in accordance with the instrument's operating instructions. The reagent blank shall be read against distilled water. A high absorbance for the reagent indicates contamination of reagent blank or improper solution preparation. If absorbance for the reagent blank compared to distilled water is above 0.040 (using a ~~12-mm~~ 12 mm cell path length) or above 0.030 (using a ~~10-mm~~ 10 mm cell path length), repeat the entire standardization procedure.

8.3.3 Zero the instrument on the reagent blank, or leave the instrument zeroed on distilled water, and subtract the absorbance of the reagent blank from the absorbance of the sample solutions.

8.3.4 Read and record absorbance at 580 nm of each sample prepared (see 9.1 for calculation).

8.3.5 When a precise desiccator value is required and the sample solution is found to fall outside the stated absorbance range (greater than 1.0 or as determined in A3.9), repeat 8.2.1 – 8.3.4. Otherwise, report the desiccator value associated with a greater than 1.0 absorbance value. When 8.2.1 – 8.3.4 are repeated, appropriately dilute the sample solution to fall within the preferred absorbance range of the spectrophotometer. Make dilution by pipetting x mL of text solution to (4-x mL) of distilled water for a total of 4 mL (that is, 1 mL of test solution + 3 mL distilled water = 4 mL total). Rerunning the distilled water "blank" is not required. Use average sample determinations as the sample absorbance. Read micrograms (µg) of formaldehyde from the calibration curve. (See Annex A3.)

9. Calculation

9.1 Calculate formaldehyde concentration in weight per unit volume in the solution from the petri dish aliquot in the desiccator:

$$c_t = \frac{c_s}{D \times 4} \tag{1}$$

where:

c_t = µg of formaldehyde per mL of sampled solution,
~~c_s = µg of formaldehyde in 4-mL aliquot of sample read from calibration curve, and~~
c_s = µg of formaldehyde in 4 mL aliquot of sample read from calibration curve, and
D = dilution factor, for example:

$$\frac{1 \text{ mL } (original\ volume)}{4 \text{ mL } (final\ volume)} D = 0.25 \tag{2}$$

If no dilution is made, D = 1.

9.2 When the temperature at which the test is conducted differs from 24 by ¼ ~~°C~~ °C (75 by ½ ~~°F)~~ °F) or more, adjust the desiccator value obtained to a standard temperature of ~~24°C (75°F)~~24 °C (75 °F) using a formula developed by Berge et al.[9] Annex A1 contains a table of conversion factors for use at different observed test temperatures as calculated using this formula. The observed test temperature is the average temperature for the total 2-h test period.

[9] Berge, A., Mellagaard B., Hanetho, P., and Ormstad, E., *Formaldehyde Release from Particleboard—Evaluation of a Mathematical Model*, Holz Als Roh-und Werkstoff 38, 1980, pp. 252–255.

10. Report

10.1 Report the following information:

10.1.1 Test number,

10.1.2 The manner in which samples were shipped or stored, or both: wrapped separately in vapor barrier, wrapped collectively in vapor barrier, waster sheet on top and bottom, or with test surfaces unprotected,

10.1.3 Name of product manufacturer or name of company submitting sample(s), or both, and date of manufacture,

10.1.4 Description of test material to include generic product name, thickness, if surface finished or sealed (both surfaces should be described), and special treatment (if known),

10.1.5 Specimen conditioning details to include temperature (and range), relative humidity (and range), formaldehyde concentration in the air, and conditioning time to nearest hour,

10.1.6 Average room temperature during the conduct of the test (see 8.1.9),

10.1.7 Empty desiccator reading (desiccator value of distilled water in desiccator containing no samples),

10.1.8 If sample solution was read undiluted or diluted, including dilution factor,

10.1.9 Desiccator value in micrograms of formaldehyde per millilitre at test conditions; desiccator value corrected to ~~24°C (75°F),~~24 °C (75 °F), rounded to nearest 0.01 µg/mL,

10.1.10 Any deviations from the standard desiccator method shall be noted. Notations of such deviations shall include, but not be limited to, conditioning time, temperature and relative humidity during conditioning, and specimen edge wax treatment,

10.1.11 The analytical method employed if different from the modified NIOSH 3500 chromotropic acid test procedure,

10.1.12 Name and model number of spectrophotometer, and

10.1.13 Date of test.

11. Precision and Bias

11.1 Variation in the formaldehyde emission from products evaluated by this test method is a consequence of variations in the materials tested, in the conduct of the test method and the analytical procedure. Limited information does exist to show the variability expected between test results when the method is used in one or more laboratories.

11.1.1 *Repeatability (Within Laboratory)*—Test results indicate a precision of 0.06 µg/mL on the same product sample. Product manufacturing variability affects test results.[10]

11.1.2 *Reproducibility (Between Laboratory)*—A test series involving five laboratories on three matched board sets from each of two products in which temperature and relative humidity conditioning of specimens was controlled showed an average coefficient of variation of 15 % for products ranging in desiccator values from 0.31 µg/mL to 0.69 µg/mL.[10]

12. Keywords

12.1 air-borne; chromotropic acid; formaldehyde levels; small-scale; wood products

[10] *Report of Preliminary Interlaboratory Formaldehyde Large Chamber Round Robin*, Hardwood Plywood Manufacturers Association and National Particleboard Association, February 1987.

ANNEXES

(Mandatory Information)

A1. TEMPERATURE CONVERSION FACTORS FOR FORMALDEHYDE

A1.1 The following conversion table is based on the Berge et al[9] formula to correct formaldehyde concentrations for temperature:

$$C = C_o \cdot e^{-R(1/t - 1/t_o)} \quad \text{(A1.1)}$$

or:

$$C_o = C \cdot e^{R(1/t - 1/t_o)} \quad \text{(A1.2)}$$

where:

C = test formaldehyde concentration level,
C_o = corrected formaldehyde concentration level,
e = natural log base,
R = coefficient of temperature (9799),
t = actual temperature, °K, and
t_o = corrected temperature, °K.

A2. STANDARD SOLUTIONS A AND B

A2.1 Standardization of Formaldehyde Standard Solution A (1.0 mg/mL)

A2.1.1 Pipet 2.70 mL of 37.0 % formaldehyde solution into a 1 L volumetric flask. Dilute to mark with freshly distilled water and mix well. This solution is stable for at least one month in a closed container at lab conditions.

A2.1.2 Calibrate the pH meter with standard buffer solution of pH 9.0.

A2.1.3 Pipet two 50 mL aliquots of formaldehyde standard Solution A into two ~~150-mL~~ 150 mL beakers for duplicate analysis and add 20 mL of 1 *M* sodium sulfite (Na_2SO_3) to each beaker. Sodium sulfite solution can age, thus the 1 *M* sodium sulfite solution should be adjusted to 9.5 pH before adding to standard Solution A aliquots.

TABLE A1.1 Temperature Conversion Table for Formaldehyde

NOTE 1—The Berge et al equation[9] is an exponential function. The greater the variance between actual and corrected temperature, the greater the potential error. These conversion factors shall not be applied beyond the specified test temperature range of ~~24 ± 1°C (75 ± 2°F).~~24 °C ± 1 °C (75 °F ± 2 °F).

~~Actual~~ Actual		~~To Convert To 24°C (75°F) Multiply by~~ To Convert To 24 °C (75 °F) Multiply by	~~Actual~~ Actual		~~To Convert To 24°C (75°F) Multiply by~~ To Convert To 24 °C (75 °F) Multiply by
°C	(°F)		°C	(°F)	
22.00	(71.6)	1.25	24.00	(75.2)	1.00
22.25	(72.1)	1.22	24.25	(75.7)	0.97
22.50	(72.5)	1.18	24.50	(76.1)	0.95
22.75	(73.0)	1.15	24.75	(76.6)	0.92
23.00	(73.4)	1.12	25.00	(77.0)	0.90
23.25	(73.9)	1.09	25.25	(77.5)	0.87
23.50	(74.3)	1.06	25.50	(77.9)	0.85
23.75	(74.8)	1.03	25.75	(78.4)	0.82
			26.00	(78.8)	0.80